



**UNIVERSITI PUTRA MALAYSIA**

**PREPARATION AND CHARACTERISATION OF SAGO  
STARCH-GRAFTPOLY(ACRYLIC ACID) AND SAGO  
STARCH-GRAFT-POLY(METHYLACRYLATE)**

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**FSAS 2001 52**

**PREPARATION AND CHARACTERISATION OF SAGO STARCH-GRAFT-  
POLY(ACRYLIC ACID) AND SAGO STARCH-GRAFT-POLY(METHYL  
ACRYLATE)**

**By**

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**Thesis Submitted in Fulfilment of the Requirement for the Degree of Master of  
Science in Faculty of Science and Environmental Studies  
Universiti Putra Malaysia**

**February 2001**



Abstract of the thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree Master of Science.

**PREPARATION AND CHARACTERISATION OF SAGO STARCH-GRAFT-POLY(ACRYLIC ACID) AND SAGO STARCH-GRAFT-POLY(METHYL ACRYLATE)**

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**February 2001**

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Acrylic acid and methyl acrylate were grafted onto sago starch by using potassium persulphate as an initiator under nitrogen gas atmosphere. The graft yield showed dependence on amount of initiator, amount of monomer, reaction temperature and reaction period. For sago starch-g-poly(acrylic acid), the optimum conditions were as follows: reaction temperature, 40 °C; reaction period, 1.5 h; amount of initiator, 10 mmoles and amount of monomer, 0.0219 moles. However, for sago starch-g-poly(methyl acrylate), the optimum conditions obtained were reaction temperature, 50 °C; reaction period, 1.5 h; amount of initiator, 8.33 mmoles and amount of monomer, 0.087 moles. TGA studies showed that the grafted sago starches exhibit different thermal behavior as compared to sago starch. SEM micrographs showed that both

acrylic acid and methyl acrylate were grafted homogeneously onto sago starch. The sorption capacity of sago starch-g-poly(acrylic acid) for Cu(II) was determined. The biodegradability study of the grafted copolymers was carried out by using  $\alpha$  – amylase lipase. The results show that the maximum glucose produced for sago starch-g-poly(acrylic acid) was on the 2<sup>nd</sup> day of experiment. However, for sago starch-g-poly(methyl acrylate), the maximum glucose produced was on the 3<sup>rd</sup> day. Swelling behavior of sago starch-g-poly(acrylic acid) in water and various salt solutions and sago starch-g-poly(methyl acrylate) in water and solvents were also investigated. The viscosity-average molecular weight increased with increase of poly(methyl acrylate) content in the graft copolymer. Sago starch-g-poly(methyl acrylate) has also been converted into poly(hydroxamic acid) chelating ion exchange resin using *Candida Rugosa* as a catalyst. The metal ion sorption rate of the resin produced based on Cu (II) at pH 3.5, was rapid. The metal ion separations of Fe(III)-Cu(II), Fe(III)-Cd(II), Fe(III)-Cr(III), Fe(III)-Ni(II) and Fe(III)-Pb(II) were successfully carried out.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Master Sains.

**PENYEDIAAN DAN PENCIRIAN KANJI SAGU CANGKUK POLI(ASID AKRILIK) DAN KANJI SAGUCANGKUK POLI(METIL AKRILAT)**

**Oleh**

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Asid akrilik dan metil akrilat dicangkukkan pada kanji sagu dengan menggunakan kalium pesulfat sebagai pemula dalam atmosfera nitrogen. Perolehan cangkukan adalah bergantung kepada amaun pemula, amaun monomer, suhu tindak balas dan tempoh tindakbalas. Bagi kanji sagu cangkuk poli(asid akrilik), keadaan tindak balas optimum adalah seperti berikut: suhu tindak balas, 40 °C; tempoh tindak balas, 1.5 h; amaun pemula, 10 mmol dan amuan monomer, 0.0219 mol. Manakala, untuk kanji sagu cangkuk poli(metil akrilat), keadaan tindak balas optimum adalah seperti berikut: suhu tindak balas, 50 °C; tempoh tindak balas, 1.5 h; amaun pemula, 8.33 mmol dan amuan monomer, 0.087 mol. Analisis TGA menunjukkan bahawa hasil cangkukan kanji sagu mempunyai sifat-sifat terma yang berbeza berbanding dengan kanji sagu. Mikrograf

SEM memperlihatkan asid akrilik dan metil akrilat telah dicangkukkan secara homogeneous ke atas kanji sagu. Kapasiti serapan ion logam Cu(II) bagi kanji sagu cangkuk poli(asid akrilik) telah dikaji. Biodegarasi kopolimer-kopolimer cangkukan dikaji dengan menggunakan lipase  $\alpha$ -amilasi. Keputusan menunjukkan glukosa maksimum yang dihasilkan adalah pada hari ke 2 bagi kanji sagu cangkuk poli(asid akrilik). Manakala bagi kanji sagu cangkuk poli(metil akrilat), glukosa maksimum dihasilkan pada hari ke 3. Sifat-sifat pengembangan kanji sagu cangkuk poli(asid akrilik) dalam air dan larutan-larutan garam serta kanji sagu cangkuk poli(metil akrilat) dalam air dan pelarut-pelarut organik telah dikaji. Purata berat molekul kelikatan didapati bertambah dengan pertambahan kandungan poli(metil akrilat) dalam kopolimer. Kanji sagu cangkuk poli(metil akrilat) telah ditukarkan kepada penukar ion pengkelat poli(asid hidrosamik) dengan menggunakan *Candida Rugosa* sebagai mangkin. Kadar serapan ion Cu(II) pada pH 3.5 adalah cepat. Pemisahan pasangan ion logam Fe(III)-Cu(II), Fe(III)-Cd(II), Fe(III)-Cr(III), Fe(III)-Ni(II) and Fe(III)-Pb(II) telah berjaya dilakukan.

## **ACKNOWLEDGEMENTS**

Firstly, I would like to take this opportunity to express my deepest appreciation and gratitude to supervisor Professor Dr. Wan Md. Zin Wan Yunus and co-supervisor, Associate Professor Dr. Md. Jelas Haron and Dr. Sidek Silong for their continuous supervision, invaluable advice and constructive criticisms throughout the course of study.

A special thank also goes to technical staff of the chemistry department especially Mr. Kamal Margona, Kak Rus, Mr. Jegan, Mr. Nazri and Mr. Ismail for their sincere help. I would also like to thank the Graduate School UPM and Ministry of Science, Technology and Environment for their sponsorship of PASCA.

Last but not least, I would like to thank my lab mate, Mrs. Rafeadah Rusli and Dr. Luftor Rahman and friends from Lab 401 (Ms. Chew, Ms. Halila, Mr. Zainal, Ms. Shila and Ms. Saliza), and my family members for their concern, inspiration and support to make my study here a successful one.

I certify that an Examination Committee met on 22<sup>nd</sup> February 2001 to conduct the final examination of Yiam Chong Bin on his Master of Science thesis entitled "Preparation and Characterisation of Sago Starch-Graft-Poly(Acrylic Acid) and Sago Starch-Graft-Poly(Methyl Acrylate)" in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

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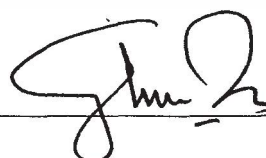
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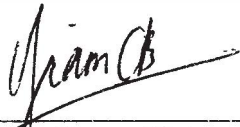
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## DECLARATION

I hereby declare the thesis is based on my origin work except for quotations and citations, which have been duly acknowledged. I declare that thesis has not been previously or concurrently submitted for any other degree at UPM or other institutions.

  
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## CHAPTER I

### INTRODUCTION

#### Starch and Sago Starch

Starch is one of the cheapest and renewable natural polymers. It occurs in living plants in the form of discrete granules ranging from about 5 – 40µm in diameter. Starch is a mixture of two polymers, amylose, a linear 1,4- $\alpha$ -D-glucose linked and amylopectin a highly branched molecule of 1,4- $\alpha$ -D-glucose with 1,6- $\alpha$ -D-glucose branches (Kennedy et al., 1983). Amylose, has a molecular weight of several hundred, while the molecular weight of branched amylopectin is of the order of several million. The ratio of amylose to amylopectin varies with the source of starch but is typically 20: 80 (Orford et al., 1987).

Starch granules contain regions of crystallinity. The granule is made up of stacks of amorphous and semi-crystalline growth rings with the thickness of 120-400 nm. The semi-crystalline shells are composed of alternating crystalline and amorphous lamellae repeating in 9- 10 nm and superimposed to the architecture of amylopectin (French, 1984). Starches from different sources differ in structure through size distribution of the granules, shape, amylose and lipid content, distribution of chain length in amylopectin and crystalline structure (Fasihuddin et al.,

1999). If the starch is slurried in water at room temperature, its solubility is negligible, even though it is a hydrophilic polymer. Starch in this form is designated as unswollen. When the water slurry is heated, hydrogen bonds of the starch are broken, a measurable fraction of the starch is dissolved and the granules swell and lose their birefringence. If the temperature of the water slurry is raised near the boiling point, the granules will largely disintegrate to form a smooth dispersion.

Sago starch is isolated from sago palm (*Metroxylon spp.*) that commonly known as 'rumbia' and distributed throughout South East Asia. Some of the important species widely used in sago starch production includes *M. longispinum*, *M. sylvestre*, *M. microcanthum*, *M. sagu* and *M. rumphii*.

The sago starch is cheap and has some other important properties such as easy to gelatinise, it has high viscosity if properly extracted and can be moulded. It has also been reported to undergo little syneresis (Takahashi, 1986).

The size of the sago starch granules can affects the viscosity of sago paste and behaviour of gelatinisation. A larger starch granule will result in rapid increase of viscosity and higher maximum viscosity. A larger starch granule also has a lower degree of gelatinisation (Radley, 1976). Sago starch granule is medium sized in the range of 15µm to 65µm (Moss, 1976). The sago starch has a gelatinisation temperature of 70 °C. Its viscosity rises rapidly during gelatinisation and decrease after its maximum viscosity. The pattern of maximum viscosity is important in



designing the force use in stirring and transporting starch paste. The stability in the viscosity of sago starch is important in ensuring the stable paste and gel during processing.

### Graft Copolymerisation

Graft copolymerisation results from the formation of an active site at a point on a polymer molecule other than its end, and exposure to a second monomer. The newly formed polymer is covalently bonded or grafted onto the existing backbone polymer molecules (Figure 1):

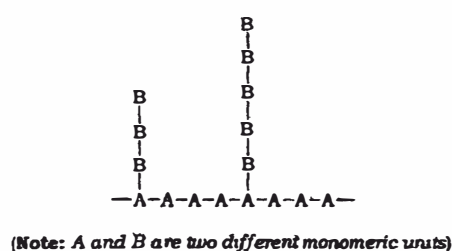


Figure 1: Structure of a Grafted Polymer